



In operando studies of an yttria stabilized zirconia electrolyte supported symmetric solid oxide cell by Dark field X-ray Microscopy at ID06

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In operando studies of an yttria stabilized zirconia electrolyte supported symmetric solid oxide cell by Dark field X-ray Microscopy at ID06



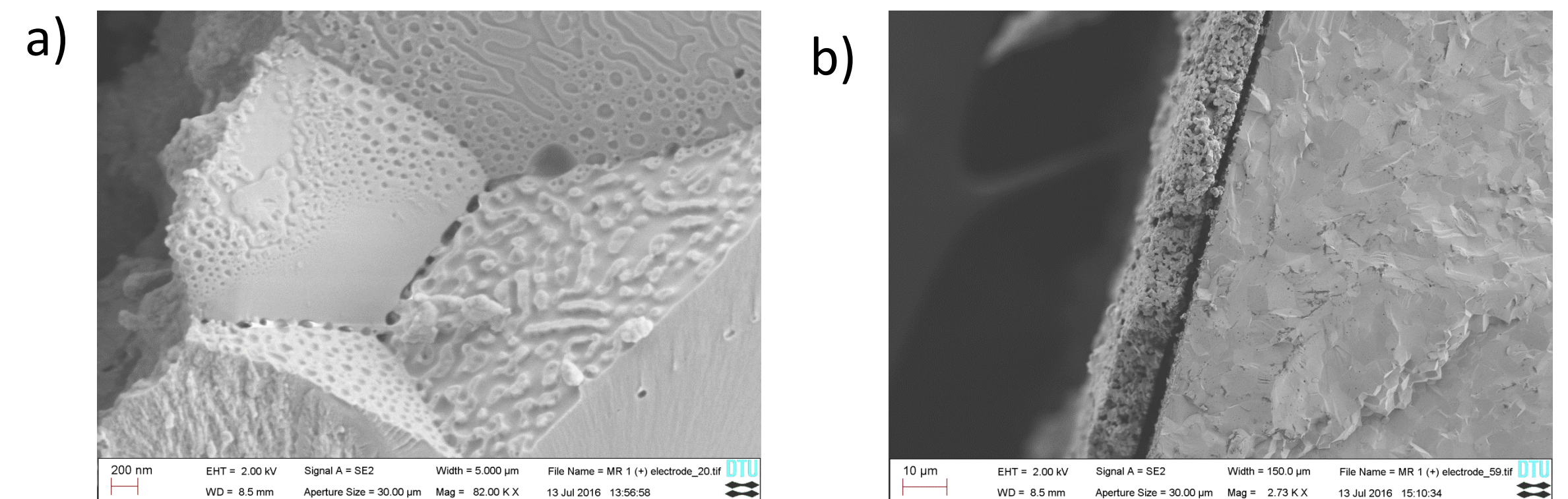
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At ID06 beamline we are currently commissioning a dark field microscope, enabling to zoom into mm-sized samples and perform 3D mapping of grains and stresses at the 100 nm scale from local regions. This provides unprecedented opportunities for studying microstructural changes in operando materials. Yttria Stabilized Zirconia is a well-known material used as high temperature electrolyte in solid oxide cells for sustainable and renewable power generation. Oxygen bubble formation at grain boundaries of YSZ near the electrolyte/oxygen electrode interface has been observed as a degradation process of electrolyser cells running at extreme operating conditions.

SEM images. a) Bubble formation in the YSZ grain boundaries, near the electrode/electrolyte interface). b) Delamination of the positive electrode after 72 hours running at 900 °C and a potential of 2V.

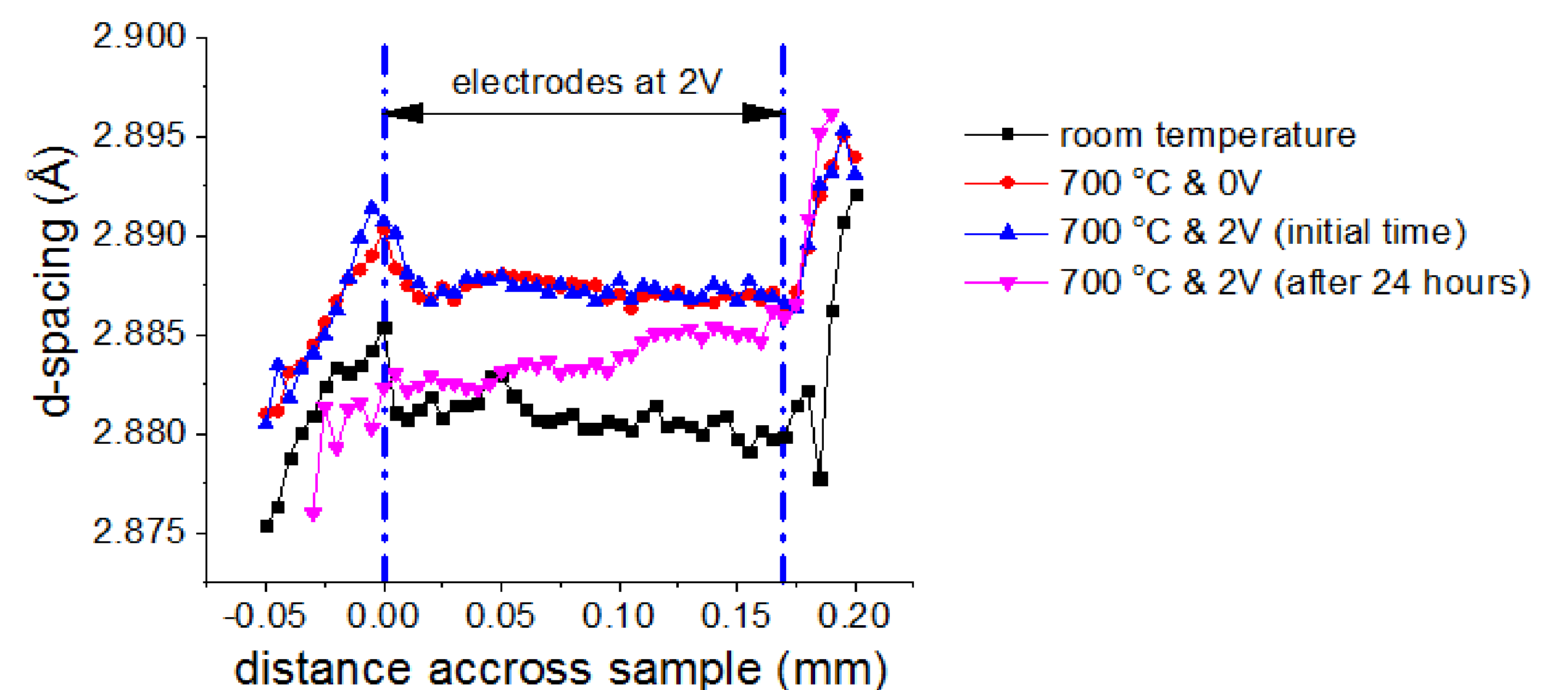
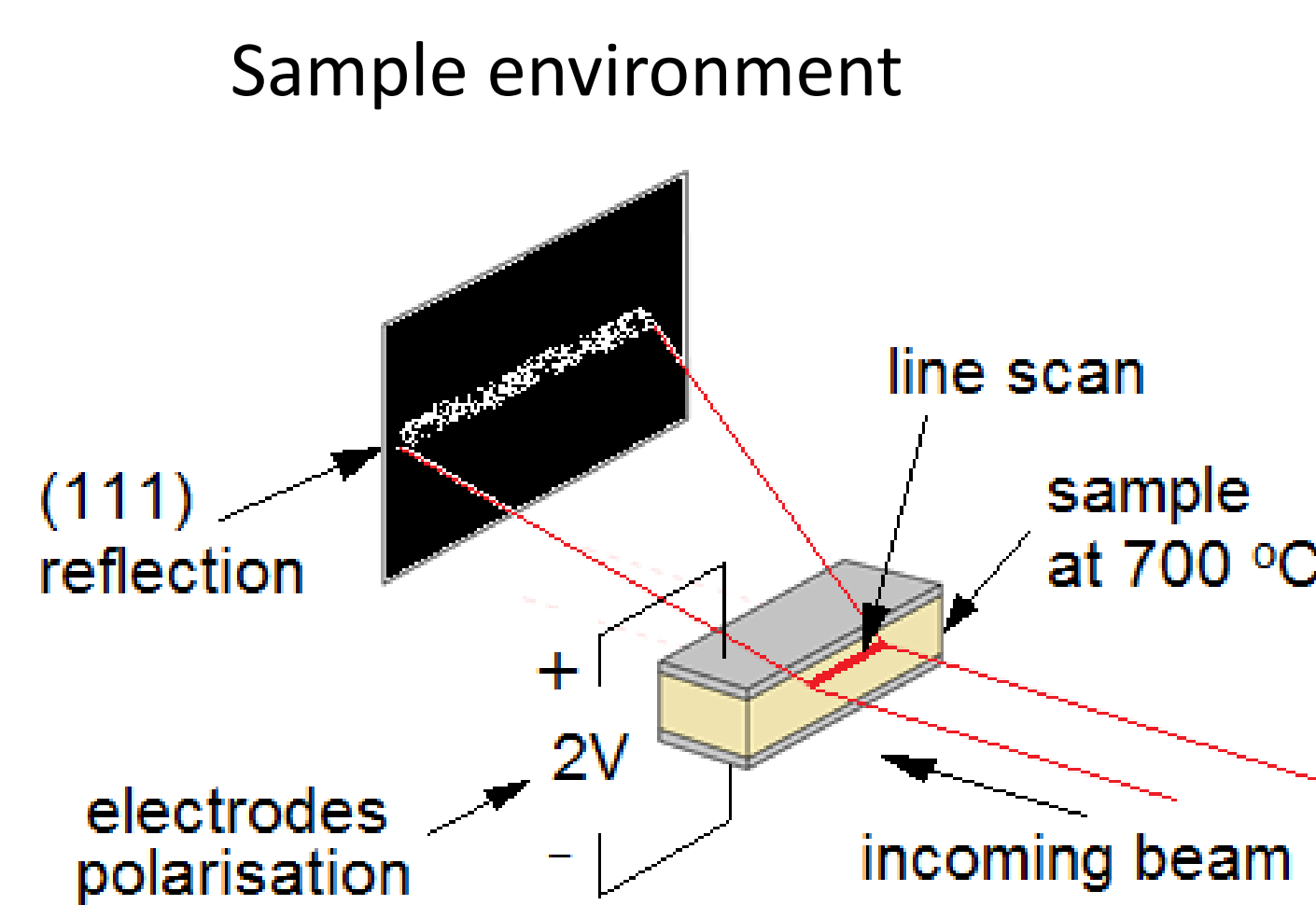
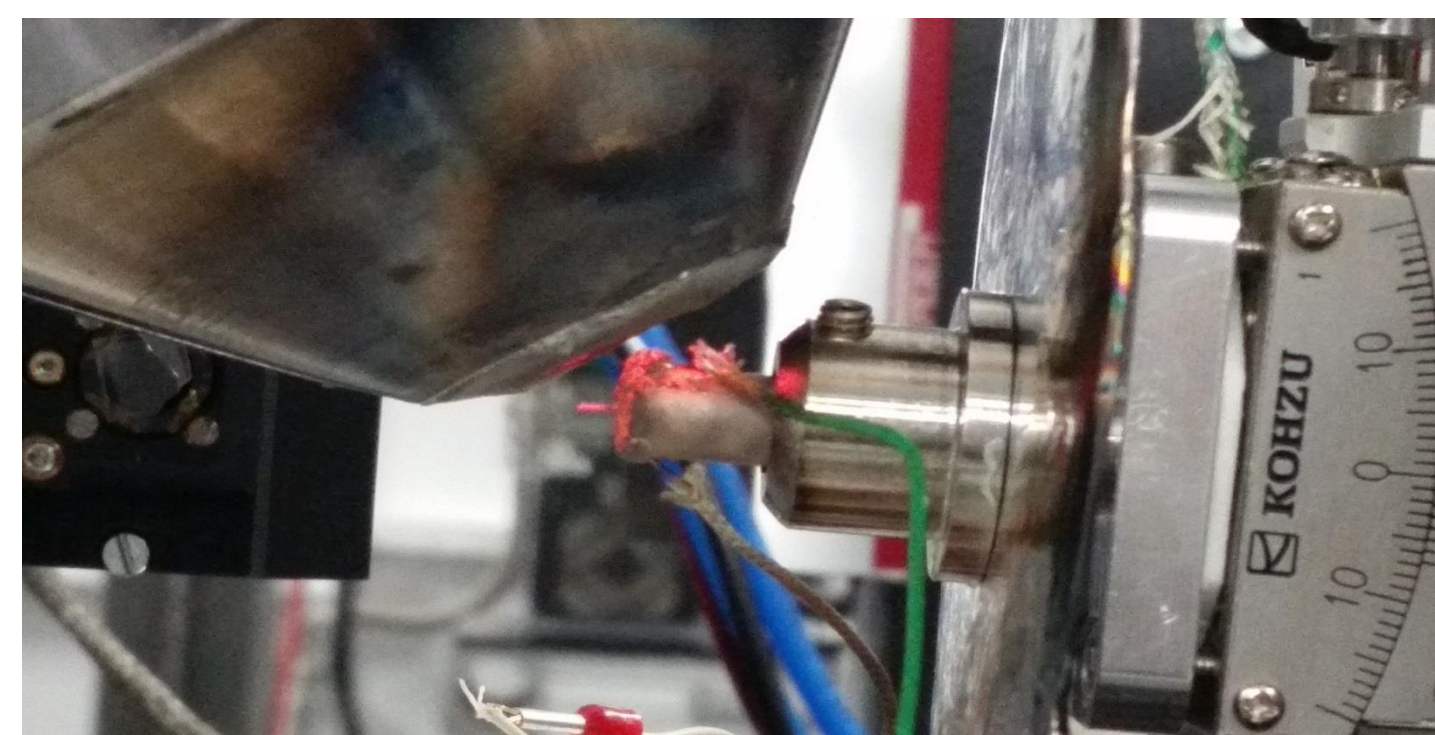


Using X-ray diffraction, the sample was scanned by a 6 microns FWHM beam in layers across, under 700 °C and a potential of 2V.

Using the YSZ (111) reflection we studied the evolution of the lattice parameter as a function of the distance to the electrodes

No immediate change in lattice parameter when applying voltage at 700 °C (lines red and blue). After 24 hours (line magenta) a gradient in lattice parameter is observed in the electrolyte, suggesting a compressive stress near the positive electrode/electrolyte interface

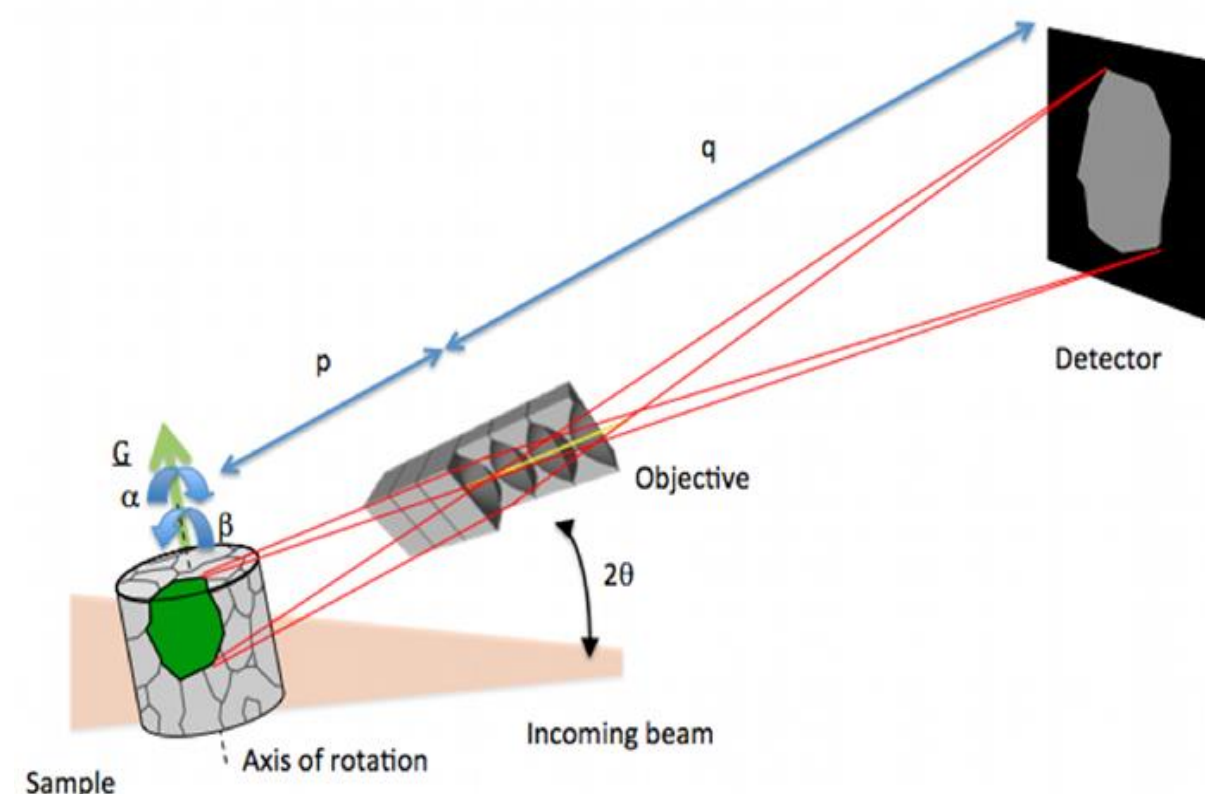
Setup of the synchrotron beamline hutch at ESRF: ID06



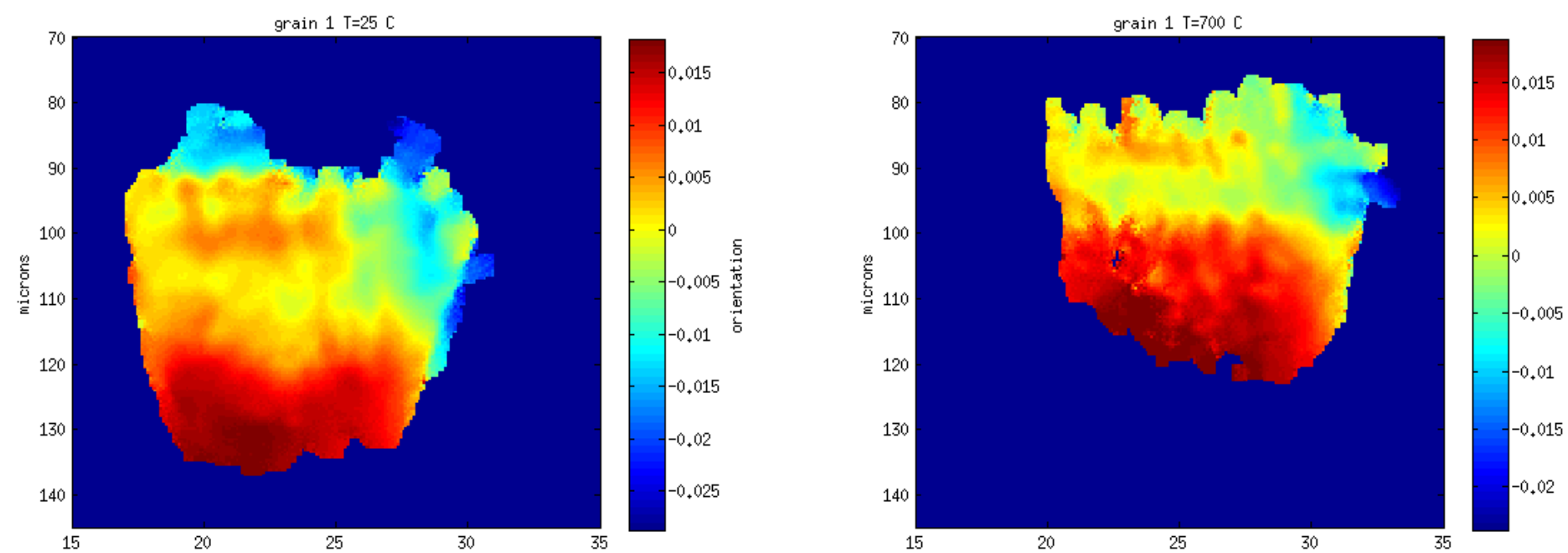
Current methods for microstructural mapping, such as Electron Back-Scatter Diffraction (EBSD) and Transmission Electron Microscopy (TEM) are inherently two-dimensional and can therefore not directly map the microstructure of bulk materials. X-ray techniques such as 3D X-Ray Diffraction (3DXRD) and Diffraction Contrast Tomography (DCT) can provide 3D maps of polycrystals, but cannot yet resolve nano-scale features.

DFXRM enables to “zoom in” by a set of lenses in the objective, generating a magnified high resolution 3D map. Dark Field X-Ray Microscopy is a promising technique for studying polycrystalline materials in 3D and is highly suited to in-situ studies of materials and their dynamics.

Principle of the dark field x-ray microscope

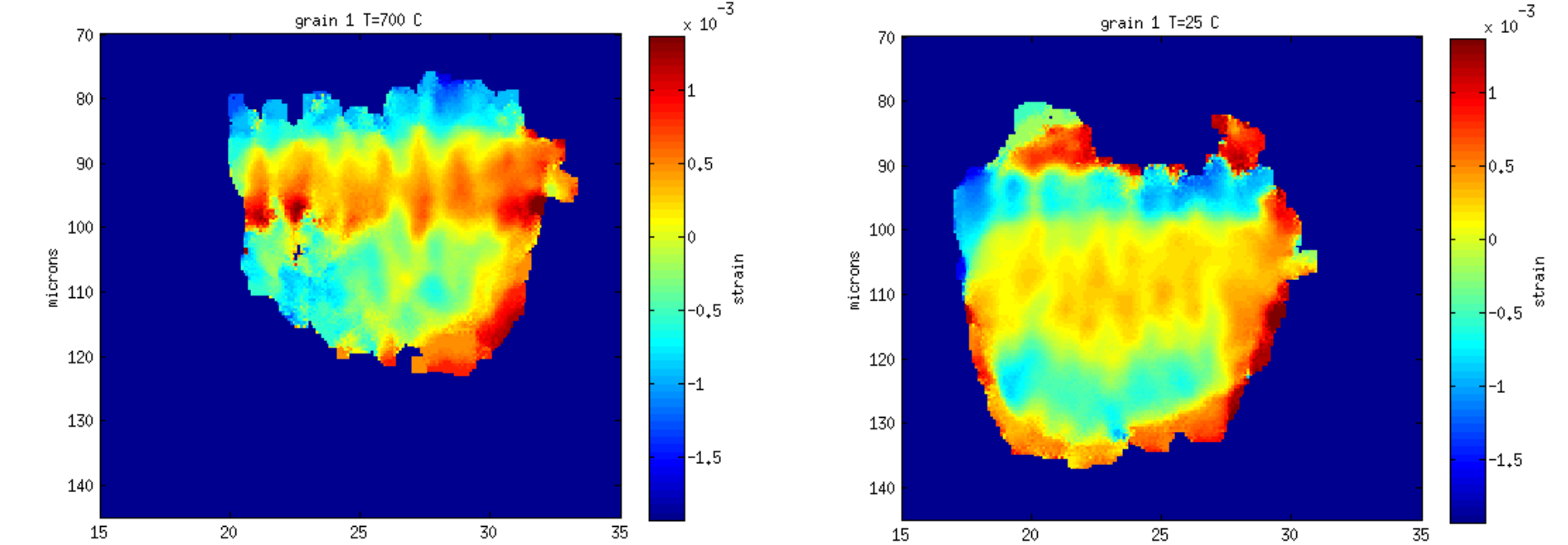


Orientation map



Mosaicity plot, where orientation is encoded with color

Strain map



Map of the average intensity of each image as a function of its angle “u” and “v” (average mosaic spread in the grain)

Dark field x-ray microscopy revealed stresses in a grain from the positive electrode/electrolyte interface and changes in the shape in the boundaries region.

References

- H. Simons, A. King, W. Ludwig, C. Detlefs, W. Pantleon, S. Schmidt, F. Stöhr, I. Snigireva, A. Snigirev and H. F. Poulsen. Nature Communications. DOI: 10.1038/ncomms7098. 2015
- R. Knibbe, M. L. Traulsen, A. Hauch, S. D. Ebbesen and M. Mogensen. Journal of The Electrochemical Society, 157 (8) B1209-B1217 2010
- F. Tietz, D. Sebold, A. Brisse, J. Schefold. Journal of Power Sources, 223 (2013) 129-135
- A. Hagen, M. L. Traulsen, W. Kiebach, B. S. Johansen. Journal of Synchrotron Radiation (2012). 19, 400-407. DOI: 10.1107/S0909049512006760

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